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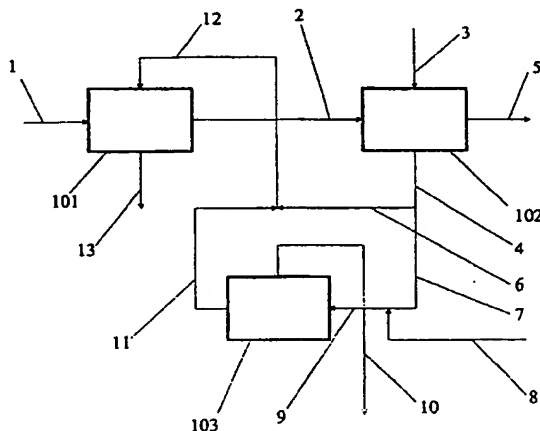
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(54) Title: PURIFICATION OF ALKALINE WASHING LIQUID



(57) Abstract: A method for washing sulphate cellulose pulp in the washing department of a sulphate cellulose mill, in which method a first pulp flow (1) is conducted to a first washer (101), where a washing liquid (12) is combined with the first pulp flow (1). A second pulp flow (2) is conducted from the first washer (101) to a second washer (102), where a washing liquid (3) is combined with the second pulp flow (2). The washed pulp flow (5) is proceeded from the second washer (102) in the process and the filtrate (4) displaced by the washing liquid (3) is recycled to the process so that the filtrate (4) displaced by the washing liquid (3) in the second washer (102) is divided into a first part flow (6) and a second part flow (7) and the pH of the second part flow (7) is dropped by combining an acidifier (8) with the second part flow (7). The acidified part flow (9) is conducted to a process unit (103), where sludge (10) containing extractives and metals is separated from the part flow (9) and the entire process and the part flow (11) treated in the process unit (103) is combined with the first part flow (6) and is recycled to the process for use as the washing liquid (12) in the first washer (101).

### **Purification of alkaline washing liquid**

The invention relates to a method for washing an alkaline liquid. More specifically, the invention relates to a method for removing extractives and metals from an alkaline liquid in the washing department of a sulphate cellulose mill.

In the production of sulphate cellulose pulp, new fibre lines with voluminous outputs have involved the problem of the detrimental effect of substances derived from pitch and various metals on pulp production as fibre lines are increasingly closed and water amounts decreased. Extractives cause detrimental adhesions to the equipment surfaces. Substances derived from pitch also cause runnability and quality problems in paper and board machines. Under propitious conditions, some metals form deposits and are thus harmful to the pulp production process.

It is previously known that extractives and metals can be removed by flocculation or precipitation. These known methods have more efficient operation in neutral or acidic conditions. However, dropping the pH to the neutral or acidic range during the washing of sulphate cellulose pulp results in undesired reactions of residual lignin, and these hamper practical implementation of known methods.

There are several known methods for washing an alkaline liquid. One of these is disclosed in WO patent specification 88/04705. This specification discloses washing of alkaline fibrous pulp in several steps and dropping the pH in at least one wash step to a level low enough for the detrimental substances to be removed from the fibrous pulp by means of any known method. However, the method disclosed in this reference involves the undesired reactions caused by the pH drop of the pulp mentioned above.

FI patent specification 52876 discloses a second method. This method comprises washing of cellulose pulp in a multi-stage washing plant, the pH of the pulp being decreased by acidification of the washing liquid for enhanced washing effect. The method has the purposes of brightened waste water, reduced effervescence at the screen level and an enhanced brightness degree of unbleached paper grades. This method also results in a low overall pH value and in the occurrence of undesired reactions in the pulp.

The method of the invention allows more efficient washing of sulphate cellulose pulp and efficient removal of extractives and metals without the pH level of the

main flow being too much decreased and without causing other problems in the process. The method of the invention is characterised by the features defined in the characterising part of the independent claim.

The advantages mentioned above are achieved by dividing the alkaline filtrate into two part flows, the pH level of one of these being dropped to the optimal level in terms of extractive and metal removal, and by recycling the purified part flow into the process. This maintains the pH level of the main flow at an adequately high level and the problems mentioned above do not occur. The division of this alkaline filtrate into part flows is most advantageously performed with the filtrates from the last steps of the washing department, these filtrates having low residual lignin and alkali concentrations.

The pH regulation of the alkaline filtrate proper can be carried out e.g. using carbon dioxide (CO<sub>2</sub>) or mineral acids. The use of carbon dioxide in pH regulation of a part flow does not alter the chemical balance of the process.

Preferred embodiments of the invention are characterised by the features defined in the claims below.

The invention is explained in greater detail below by means of examples and with reference to the figures, in which

Figure 1 is a flow chart of the process,

Figure 2 is a schematic view of the pulp flow division in the process of figure 1, and

Figure 3 is a table of the reductions achieved with the method.

Figure 1 shows a first pulp flow 1, which reaches a first washer 101. A second pulp flow 2 starts from the first washer 101 to the second washer 102, which may be the last brown mechanical pulp washer or any other washer of the washing department. The method is also applicable to more than one washer in a washing department.

The second pulp flow 2 is a combination of the first pulp flow 1 and the liquid 12 from the process unit 103, which are combined in washer 101. Thus the liquid 12 will act as the washing liquid of the first washer 101.

A washing liquid 3 will also enter the second washer 102, this washing liquid being combined with the second pulp flow 2 coming from the first washer 101. The alkaline filtrate displaced by the washing liquid 3 in the second washer 102 is separated from the second pulp flow 2 and the washed third pulp flow 5 is removed by proceeding in the process. If necessary, the pulp flow 5 can be subjected to any further treatment. If the alkaline filtrate 4 contains a large amount of fibres, the filtrate is advantageously filtrated before any further treatment in order to minimise fibre losses.

The alkaline filtrate 4 separated from the washed second pulp flow 2 in the second washer 102 is divided into two part flows 6 and 7. The alkaline filtrate 4 may be divided into e.g. 2/3 first part flows 6 and 1/3 second part flow 7. The second part flow 7 of alkaline filtrate 4 is further treated in the process by combining it with an acidifier 8, such as e.g. carbon dioxide (CO<sub>2</sub>) in order to drop the pH of the part flow. The pH of the second part flow 7 of the filtrate 4 can be dropped e.g. to 7 (neutral pH), which is optimal with a view to further treatment of the part flow 7. Next the part flow 9 treated with carbon dioxide is taken to process unit 103. The use of carbon dioxide in pH regulation does not alter the chemical balance of the process. Instead of carbon dioxide, one could use e.g. mineral acids as acidifiers. The first part flow 6 of the alkaline filtrate 4 proceeds untreated in the process.

The filtrate 4 from the washer 102 can be divided into part flows 6 and 7 also in any other ratio than 2:1 as above, e.g. 5:1, 4:1, 3:1 or 1:1. The division into part flows 6 and 7 in the process has a direct impact on the amount of sludge 10 to be removed and thus of detrimental substances.

In process unit 103, extractives, metals and other detrimental substances are removed from part flow 9 with the use of any methods known *per se* (coagulation, flocculation, flotation or the like). When the pH of part flow 9 is approx. 7, the optimal situation in terms of e.g. flotation has been achieved in process unit 103. The sludge 10 produced during the treatment, which contains the detrimental substances, is removed from process unit 103. The purified part flow 11 is combined after process unit 103 with the first part flow 6 of the alkaline filtrate 4, producing washing liquid 12, which is taken to washer 101. The liquid produced by recombination of the first part flow 6 and the second part flow 7 of the alkaline filtrate 4 is consequently recycled to the process and it is used as the washing liquid 12 of the first washer. Addition of washing liquid 12 to the pulp flow 1 in washer 101 does not drop the pH of the pulp flow substantially nor does it cause problems,

such as undesired reactions of residual lignin. Similarly, washing liquid 12 displaces filtrate 13 in washer 101, and filtrate 13 can be treated in a manner similar to that of filtrate 4 of washer 102, if desired.

Figure 2 shows an example of a preferred option of washing sulphate cellulose pulp. The pulp flow 2 reaching the washer 102 is illustrated with fibres 21 and liquid 22 as separate parts. The pulp flow 2 entering washer 102 contains 6.80/Adt of substances to be removed. The filtrate 4 displaced by the washing agent from washer 102 contains 4.20 kg/Adt of substances to be eliminated and the washed pulp flow 5 2.60 kg/Adt accordingly. The fibres of the pulp flow 5 then contains about 0.28% of extractives. In this example, 1/3 of the filtrate is treated in process unit 103, the liquid 9 reaching this unit containing 1.388 kg/Adt of substances to be removed. After the treatment, 75% of the substances reaching the process unit 103 are removed from the process, i.e. 1.04 kg/Adt as indicated by arrow 10. The untreated portion 6 (2/3) of the filtrate 4, which contains 2.814 kg/Adt of detrimental substances, is combined with the portion 11 purified in process unit 103 and is recycled to the washer 101. In this example, the washing liquid 12 of washer 101 contains 3.16 kg/Adt of detrimental substances.

Next follows a practical example of the treatment of an alkaline filtrate of birch pulp at a sulphate cellulose mill with the method of the invention by means of the reduction percentages thus achieved. In the first case, the part flow 7 of filtrate 4 has been acidified to reduce its pH to 9.4, and in the second case, to 7.0, whereas the original filtrate 4 had a pH > 10. The results achieved with the method of the invention are summarised in the table of figure 3. With pH 9.4 of the liquid, the reduction percentages were 0% for fatty acid, 13% for resinic acid, 30% for calcium and 21% for magnanum, respectively. Accordingly, with the pH of the liquid dropped to 7.0, the reduction percentages were 86% for fatty acid, 66% for resinic acid, 69% for calcium and 75% for magnanum. The reduction percentages have been calculated on the part flow passing through process unit 103 (incoming flow 9 and outgoing flow 11) with the flotation method used for purification in the process unit.

The purpose of the method exemplified above is to maintain the pH of the pulp main flow and to remove detrimental substances from the process merely by dropping the pH of a part flow of the filtrate to a low level, and to recycle the purified filtrate to the main flow. Dropping the pH of a small part flow alone does not have a substantial impact on the pH value of the entire pulp flow, and hence it is

possible to avoid problems relating e.g. to residual lignin occurring in known methods.

## Claims

1. A method for washing an alkaline liquid in the washing department of a sulphate cellulose mill, the method comprising the following steps in the order below:

- conducting a first pulp flow (1) to a first washer (101), where a washing liquid (12) is combined with the first pulp flow (1),
- conducting a second pulp flow (2) from the first washer (101) to a second washer (102), where a washing liquid (3) is combined with the second pulp flow (2),
- proceeding the washed pulp flow (5) from the second washer (102) in the process and recycling the filtrate (4) displaced by the washing agent (3) to the process,

**characterised in**

- dividing the filtrate (4) displaced by the washing agent (3) from the washer (102) into a first part flow (6) and a second part flow (7),
- decreasing the pH of the second part flow (7) by combining an acidifier (8) with the second part flow (7)
- conducting the acidified part flow (9) to the process unit (103), where the sludge (10) containing extractives and metals is removed from the part flow (9) and the entire process
- combining the part flow (11) treated in the process unit (103) with the first part flow (6) and recycling it to the process for use as the washing liquid (12) of the first washer (101).

2. A method as defined in claim 1, **characterised** in that the filtrate (4) from the washer (102) is divided into a first part flow (6) and a second part flow (7) in the ratio 1:1, 2:1, 3:1, 4:1 or 5:1.

3. A method as defined in claim 2, **characterised** in that the pH of the second part flow (7) of the filtrate (4) is decreased to the range 6 to 8.

4. A method as defined in claim 2, **characterised** in that the pH of the second part flow (7) of the filtrate (4) is decreased to the range 6.5 to 7.5.

5. A method as defined in claim 3 or 4, **characterised** in that carbon dioxide, mineral acids or other similar substances are used as an acidifier (8).

6. A method as defined in claim 5, **characterised** in that the washer (101) is the one before the last and the washer (102) is the last washer.

7. A method as defined in claim 6, **characterised** in that the extractives and metals are removed from the process unit (103) by coagulation, flocculation and flotation or any similar methods.



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<b>pH of the liquid</b>	<b>Fatty acid</b>	<b>Resinoic acid</b>	<b>Calcium</b>	<b>Magnanium</b>
9.4	0	13	30	21
7.0	86	66	69	75

Fig. 3